CERTIFICATE

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Signed this 14th day of September, 2007

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JAPAN PATENT OFFICE

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[Title of the Document] Specification

[Title of the Invention] METHOD OF MANUFACTURING MAGNETIC PARTICLE, MAGNETIC PARTICLE AND MAGNETIC RECORDING MEDIUM

[Claims for the Patent]

[Claim 1]

A method of manufacturing a magnetic particle, comprising:

the alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and

the magnetic particle formation step;

wherein in said alloy preparation formation step, by using a mixing and reaction device which has a stirring vane rotating at a high speed in the interior of a mixer, a plurality of kinds of solutions for preparing said alloy particle are supplied to the interior of said mixer, where the plurality of kinds of solutions are mixed together and caused to react with each other by a liquid phase process so that the peripheral speed in a leading end portion of said stirring vane is not less than 5 m/second.

[Claim 2]

A method of manufacturing a magnetic particle, comprising:

the alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and

the magnetic particle formation step;

wherein in said alloy preparation formation step, by using a mixing and reaction device in which there is provided a reaction vessel which is filled with a bulk liquid and provided therein with a mixer which has a stirring vane rotating at a high speed and is provided with an opening through which said bulk liquid is circulated to and from the interior of said reaction vessel, a plurality of kinds of solutions for preparing said alloy particle are supplied to the interior of said mixer, where the plurality of kinds of solutions are mixed together and caused to react with each other by a liquid phase process, and at the same time a mixed reaction solution is discharged from said mixer to said reaction vessel by a circulating stream of said bulk liquid.

[Claim 3]

The method of manufacturing a magnetic particle according to claim 2, wherein the peripheral speed in a leading end portion of said stirring vane is not less than 5 m/second.

[Claim 4]

A method of manufacturing a magnetic particle, comprising:

the alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and

the magnetic particle formation step;

wherein in said alloy preparation formation step, by using a mixing and reaction device which is provided with a mixer in the interior of a reaction vessel and has a microgap formed between an inner wall of the mixer and a stirring member rotating at a high speed and in which in order to form this microgap, when the distance from the center of rotation of said stirring member to a leading end thereof is put as 1, the distance to said inner wall having the shortest distance from the center of rotation of said stirring member is set in the range of 1.001 to 1.200, a plurality of kinds of solutions for preparing said alloy particle are supplied to said microgap, where the plurality of kinds of solutions are mixed together and caused to react with each other by a liquid phase process, and at the same time the mixed reaction solution is discharged from said microgap.

[Claim 5]

The method of manufacturing a magnetic particle according to claim 4, wherein the peripheral speed in a leading end portion of said stirring member is not less than 5 m/second.

[Claim 6]

The method of manufacturing a magnetic particle according to any one of claims 1 to 5, wherein said liquid phase process is the reversed micelle process and wherein as said plurality of kinds of solutions, a reversed micelle solution (solution L1), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution, and a reversed micelle solution (solutions L2), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing a plurality of kinds of metallic atoms constituting said alloy particle, are prepared, and said solution L1 and solutions L2 are supplied to said mixer.

[Claim 7]

The method of manufacturing a magnetic particle according to any one of claims 1 to 5, wherein the liquid phase process is a reversed micelle process and as the plurality of kinds of solutions, a reversed micelle solution (solution L1), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution, and a reversed micelle solution (solution L3), which is composed of a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing one of a plurality of kinds of metallic atoms constituting the alloy particle, are prepared, the number of prepared Solutions L3 being equal to the number of the plurality of kinds of metallic atoms, and the solution L1 and the plurality of solutions L3 are supplied to the mixer.

[Claim 8]

The method of manufacturing a magnetic particle according to any one of claims 1 to 7, wherein at least two kinds of metallic atoms constituting the alloy particle capable of forming said CuAu type or Cu_3Au type hard magnetic ordered alloy phase are selected from the Groups VIb and VIII of the long periodic table and at least further one kind of metallic atom is selected from the Groups I b, III a and IV a, the content of said one kind of metal atom being 1 to 30 atom % of the whole alloy.

[Claim 9]

The method of manufacturing a magnetic particle according to any one of claims 1 to 8, wherein the mixing and reaction temperature in said alloy particle preparation step is controlled to the range of -5°C to 30°C.

[Claim 10]

The method of manufacturing a magnetic particle according to any one of claims 1 to 9, wherein in said magnetic particle formation step of forming a CuAu type or Cu_3Au type magnetic particle from the alloy particle prepared in said alloy particle preparation step, annealing treatment is performed after the application of an alloy-particle-containing solution, which contains the alloy particle prepared in said alloy particle preparation step to a backing.

[Claim 11]

The method of manufacturing a magnetic particle according to claim 10, wherein the annealing treatment temperature in said annealing treatment is controlled in the range of 100°C to 500°C.

[Claim 12]

A magnetic particle manufactured by the method of manufacturing a magnetic particle according to any one of claims 1 to 11.

[Claim 13]

A magnetic recording medium containing the magnetic particle according to claim 12 in a magnetic layer.

[Detailed Description of the Invention]

[0001]

[Field of the Invention]

The present invention relates to a method of manufacturing a magnetic particle, a magnetic particle manufactured by the method and a magnetic recording medium containing this magnetic particle in a magnetic layer.

[0002]

[Conventional Art]

Reducing the particle size of a magnetic particle contained in a magnetic layer is important for increasing the magnetic recording density. For example, in magnetic recording media widely used as video tapes, computer tapes, disks, etc., noise decreases when the particle size is reduced in a case where the mass of a hard magnetic material is the same.

[0003]

A CuAu type or Cu_3Au type hard magnetic ordered alloy is attracting attention as a material for a magnetic particle which is promising for improving the magnetic recording density (described, for example, in Patent Document 1 and Patent Document 2). Because this hard magnetic ordered alloy has large crystal magnetic anisotropy because of strains generated during ordering and it is known that this alloy shows hard magnetism even when the particle size of a magnetic particle is reduced.

[0004]

Although a magnetic particle showing hard magnetism is prepared by a liquid phase process, a gaseous phase process, etc., a magnetic particle immediately after the preparation of a practical liquid phase process excellent in mass producibility, in particular, has a structure of an unordered face-centered cubic crystal. A face-centered cubic crystal usually shows soft magnetism or paramagnetism and is unsuitable for a magnetic recording medium. In a magnetic recording medium, therefore, it is necessary to obtain a hard magnetic ordered alloy having a coercive force of not less than 95.5 kA/m (1200 Oe). In order to obtain such a hard magnetic ordered alloy, it is necessary to cause an unordered phase to transform to an ordered phase. For this reason, the manufacturing of a magnetic particle comprises the alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and the magnetic particle formation step of forming a CuAu type or Cu_3Au type magnetic particle from the alloy particle formed in this alloy particle preparation step, and in the magnetic particle formation step, usually annealing treatment (heat treatment) is performed.

[0005]

However, whether the performance of a magnetic particle used in a magnetic recording medium is good is not determined by the magnetic particle formation step alone, and the key point is how to prepare, in the alloy particle preparation step, an alloy particle which has a micro size, is excellent in monodispersibility and provides an excellent transformation facilitativeness which facilitates the transformation of an unordered phase to an ordered phase. Usually, the preparation of an alloy particle is performed by mixing a plurality of kinds of solutions for alloy particle preparation by the above-described liquid phase process, and a device in which a stirring

vane is provided in a tank as shown in Figure 9 has been proposed as a mixing and reaction device (Patent Document 3).

[0006]

In this mixing and reaction device 1, in order to stir solutions filled in the interior of a tank 2, a stirring vane 5 is rotated and driven via transmission device 4 which transmits the rotational driving force of a motor 3 in a noncontact manner by use of magnetic force. On the outer circumferential surface of the tank 1 is provided temperature control device 6, which heats or cools the solutions in order to control the temperature control of the solutions filled in the interior. A sealing lid 7 of this tank 2 is provided with an introduction pipe 8 which introduces the solutions and the lower part of the tank 2 is provided with a discharge pipe 9 which discharges mixed reaction solutions which are mixed and caused to react within the tank 2.

[0007]

[Patent Document 1] Japanese Patent Application Laid-Open No. 2003-6830

[0008]

[Patent Document 2] Japanese Patent Application Laid-Open No. 2001-256631

[0009]

[Patent Document 3] Japanese Patent Application Laid-Open No. 5-173267

[0010]

[Problems to be Solved by the Invention]

However, in the preparation of an alloy particle by using a conventional mixing and reaction device, a dead space is present within the tank 2, making the stirring of the solution nonuniform, and particle growth occurs due to the partial circulation of a formed alloy particle within the tank 2. Therefore, the conventional preparation of an alloy particle has the drawback that it is impossible to produce an alloy particle which has a micro size, is excellent in monodispersibility and provides an excellent transformation facilitativeness.

[0011]

The present invention was made in view of such a situation and has as its object the provision of a method of manufacturing a magnetic particle, which is capable of manufacturing a magnetic particle having good performance because an alloy particle which meets all of the requirements for a micro size, monodispersibility and transformation facilitativeness can be prepared in the alloy particle preparation step, a magnetic particle manufactured by this method and a magnetic recording medium containing this magnetic particle in a magnetic layer.

[0012]

[Means for Solving the Problems]

The inventors of the present invention has obtained the knowledge that an alloy particle which has a micro size, is excellent in monodispersibility and provides an excellent self-assembling property can be obtained by mixing together a plurality of kinds of solutions for preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and causing the solutions to react with each other by an ultrahigh-speed stirring method in which the peripheral speed at a leading end of a stirring vane is not less than 5 m/second, or an in-tank mixer method in which mixing and reaction are caused to occur in a mixer provided within a reaction vessel filled with a bulk liquid and a mixed reaction solution is discharged to the bulk liquid within the reaction vessel, or a microgap mixing method in which mixing and reaction are caused to occur in a microgap formed between an inner wall of a mixer and a stirring member rotating at a high speed. The present invention was achieved based on such knowledge.

[0013]

The particle size (particle diameter) is indicated by the diameter of a circle having an area equal to the projected area of the parallel outer surfaces of a particle. That is, the projected area of a particle is obtained by measuring the area on an electron micrograph and correcting the projection magnification. By supposing a circle having an area equal to the projected area of a particle, the diameter of this circle is regarded as the circle-equivalent diameter of an alloy particle (or a magnetic particle in some cases).

[0014]

In order to achieve the above object, in claim 1 of the present invention, there is provided a method of manufacturing a magnetic particle, which comprises: the alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and the magnetic particle formation step; wherein in the alloy preparation formation step, by using a mixing and reaction device which has a stirring vane rotating at a high speed in the interior of a mixer, a plurality of kinds of solutions for preparing the alloy particle are supplied to the interior of the mixer, where the plurality of kinds of solutions are mixed together and caused to react with each other by a liquid phase process so that the peripheral speed in a leading end portion of the stirring vane is not less than 5 m/second.

[0015]

According to claim 1 of the present invention, in the alloy preparation formation step, a plurality of kinds of solutions for preparing the alloy particle are mixed together and caused to react with each other by an ultrahigh-speed stirring method at a peripheral speed of not less than 5 m/second at a leading end of the stirring vane. As a result of this, it is possible to

instantaneously and efficiently mix a plurality of kinds of solutions together and cause these solutions to react with each other. Therefore, it is possible to form an alloy particle which has a micro size and is excellent in monodispersibility. Because this alloy particle is excellent in the self-assembling property, it is possible to cause the alloy particle to undergo transformation to a CuAu type or Cu_3Au type magnetic particle in the magnetic particle forming step. Incidentally, the peripheral speed at a leading end of the stirring vane is preferably not less than 10 m/second.

[0016]

In order to achieve the above object, in claim 2 of the present invention, there is provided a method of manufacturing a magnetic particle, which comprises: the alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and the magnetic particle formation step; wherein in the alloy preparation formation step, by using a mixing and reaction device in which there is provided a reaction vessel which is filled with a bulk liquid and provided therein with a mixer which has a stirring vane rotating at a high speed and is provided with an opening through which the bulk liquid is circulated to and from the interior of the reaction vessel, a plurality of kinds of solutions for preparing the alloy particle are supplied to the interior of the mixer, where the plurality of kinds of solutions are mixed together and caused to react with each other by a liquid phase process, and at the same time a mixed reaction solution is immediately discharged from the mixer to the reaction vessel by a circulating stream of the bulk liquid.

[0017]

According to claim 2, in the alloy particle preparation step, a plurality of kinds of solutions for preparing the alloy particle are mixed together and caused to react with each other by an in-tank mixer method which involves performing mixing and reaction in a mixer provided within a reaction vessel filled with a bulk liquid and discharging a mixed reaction solution to the bulk liquid within the reaction vessel. As a result of this, because the plurality of kinds of solutions can be instantaneously and efficiently mixed together and caused to react with each other. Therefore, it is possible to prepare an alloy particle which has a micro size and an excellent monodispersibility. Because this alloy particle prepared by this in-tank mixer method is excellent in the self-assembling property, in the magnetic particle formation step, it is easy to cause the alloy particle to undergo transformation to a CuAu type or Cu_3Au type hard magnetic particle.

[0018]

In this case, as specified in claims 3, it is desirable to ensure that the peripheral speed in a leading end portion of the stirring vane is not less than 5 m/second. In this manner, by

combining the in-tank mixer method and the ultrahigh-speed stirring method, it is possible to prepare an alloy particle excellent in a micro size, monodispersibility and the self-assembling property.

[0019]

In order to achieve the above object, in claim 4 of the present invention, there is provided a method of manufacturing a magnetic particle, which comprises: the alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and the magnetic particle formation step; wherein in the alloy preparation formation step, by using a mixing and reaction device which is provided with a mixer in the interior of a reaction vessel and has a microgap formed between an inner wall of the mixer and a stirring member rotating at a high speed and in which in order to form this microgap, when the distance from the center of rotation of the stirring member to a leading end thereof is put as 1, the distance to the inner wall having the shortest distance from the center of rotation of the stirring member is set in the range of 1.001 to 1.200, a plurality of kinds of solutions for preparing the alloy particle are supplied to the microgap, where the plurality of kinds of solutions are mixed together and caused to react with each other by a liquid phase process, and at the same time the mixed reaction solution is discharged from the microgap.

[0020]

According to claim 4 of the present invention, in the alloy particle preparation step, a plurality of kinds of solutions for preparing the alloy particle are mixed together and caused to react with each other by a microgap mixing method which involves performing mixing and reaction in a microgap formed between an inner wall of the mixer and a stirring member rotating at a high speed.

[0021]

When the distance from the center of rotation of the stirring member to a leading end thereof is put as 1, the microgap refers to a gap formed by setting the distance to the inner wall having the shortest distance from the center of rotation of the stirring member in the range of 1.001 to 1.200. The stirring member may have the shape of a cylindrical drum or may be constituted by disk-shaped stirring members which are provided in multiple stages on a rotary shaft.

[0022]

As a result of this, because due to the shearing force in the microgap the plurality of kinds of solutions can be instantaneously and efficiently mixed together and caused to react with each other. Therefore, it is possible to prepare an alloy particle which has a micro size and an

excellent monodispersibility. Because the alloy particle prepared by this microgap mixing method is excellent in the self-assembling property, in the magnetic particle formation step, it is easy to cause the alloy particle to undergo transformation to a CuAu type or Cu_3Au type hard magnetic particle.

[0023]

In this case, as described in claim 5, it is preferred that the peripheral speed in a leading end portion of the stirring member be not less than 5 m/second. In this manner, by combining the microgap mixing method and the ultrahigh-speed stirring method, it is possible to prepare an alloy particle excellent in a micro size, monodispersibility and the self-assembling property.

[0024]

Claim 6 of the present invention is that in, any one of claims 1 to 5, the liquid phase process is the reversed micelle process and that as the plurality of kinds of solutions, a reversed micelle solution (solution L1), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution, and a reversed micelle solution (solutions L2), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing a plurality of kinds of metallic atoms constituting the alloy particle, are prepared, and the solution L1 and solutions L2 are supplied to the mixer.

[0025]

According to claim 6 of the present invention, by performing the liquid phase process by the reversed micelle method, it becomes easy to control the particle diameter of a prepared alloy particle. Furthermore, it is possible to prepare, as the plurality of kinds of solutions, Solution L1 which is constituted by a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution and Solution L2 which is constituted by a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing a plurality of kinds of metallic atoms constituting the alloy particle. That is, it is possible to cause all the plurality of kinds of metallic atoms constituting the alloy particle to be contained in the solution L2 which is mixed with the solution L1 and caused to react.

[0026]

Claim 7 of the present invention is that, in any one of claims 1 to 5, the liquid phase process is a reversed micelle process and as the plurality of kinds of solutions, a reversed micelle solution (solution L1), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution, and a reversed micelle solution (solution L3), which is composed of a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing one of a plurality of kinds of metallic atoms constituting the alloy particle, are

prepared, the number of prepared Solutions L3 being equal to the number of the plurality of kinds of metallic atoms, and the solution L1 and the plurality of solutions L3 are supplied to the mixer.

[0027]

According to claim 7 of the present invention a plurality of the solutions L3 are prepared, each of which contains one kind selected from the plurality of kinds of metallic atoms constituting the alloy particle, and the solution L1 and the plurality of solutions L3 are supplied to the mixer.

[0028]

Claim 8 of the present invention is that, in any one of claims 1 to 7, at least two kinds of metallic atoms constituting the alloy particle capable of forming the CuAu type or Cu_3Au type hard magnetic ordered alloy phase are selected from the Groups VI b and VIII of the short periodic table and at least further one kind of metallic atom is selected from the Groups I b, III a and IV a, the content of the one kind of metal atom being 10 to 30 atom % of the whole alloy.

[0029]

According to claim 8 of the present invention, by adding at least one kind of metallic atom selected from the Groups I b, III a and IV a to at least two kinds of metallic atoms selected from the Groups VI b and VIII of the long periodic table, it is possible to lower the transformation temperature in causing the transformation of the alloy phase prepared in the alloy particle preparation phase to occur from an unordered phase to an ordered phase in the magnetic particle formation step.

[0030]

Claim 9 of the present invention is that, in any one of claims 1 to 8, the mixing and reaction temperature in the alloy particle preparation step is controlled to the range of -5°C to 30°C.

[0031]

According to claim 9 of the present invention, the mixing and reaction temperature in the alloy particle preparation step is controlled to the range of -5°C to 30°C. If the mixing and reaction temperature is less than -5°C, this poses the problem that a water phase condenses, making a reduction reaction nonuniform. If the mixing and reaction temperature exceeds 30°C, coalescence and precipitation tend to occur and the system may sometimes become unstable. The mixing and reaction temperature is preferably in the range of 0°C to 25°C and especially preferably in the range of 5°C to 25°C.

[0032]

Claim 10 of the present invention is that, in any one of claims 1 to 9, in the magnetic particle formation step annealing treatment is performed after the application of an alloy-particle-containing solution, which contains the alloy particle prepared in the alloy particle preparation step, to a backing.

[0033]

Although the alloy particle prepared in the alloy particle preparation step has weak magnetism, it is necessary to cause the alloy phase of the alloy particle to undergo transformation from an unordered phase to an ordered phase in order to obtain a CuAu type or Cu_3Au type hard magnetic ordered alloy having a coercive force of not less than 1200 Oe, which is required in a magnetic recording medium. However, if this annealing treatment is performed in the state of a particle, alloy particles are apt to coalesce together.

[0034]

According to claim 10 of the present invention, annealing treatment is performed after the application of an alloy-particle-containing solution, which contains the alloy particle prepared in the alloy particle preparation step, to a backing. Therefore, it is possible to prevent the coalescence of alloy particles and it is possible to form an alloy particle having a micro size.

[0035]

Claim 11 of the present invention is that, in claim 10, the annealing treatment temperature in the annealing treatment is controlled to the range of 100°C to 500°C. The annealing treatment temperature is more preferably in the range of 150°C to 500°C.

[0036]

In claim 12 of the present invention, there is provided a magnetic particle manufactured by the method of manufacturing a magnetic particle according to any one of claims 1 to 11, and in claim 13 of the present invention, there is provided a magnetic recording medium containing the magnetic particle according to claim 11 in a magnetic layer.

[0037]

[Embodiments of the Invention]

Preferred embodiments of a method of manufacturing a magnetic particle, a magnetic particle and a magnetic recording medium related to the present invention will be described below with reference to the attached drawings.

[0038]

A method of manufacturing a magnetic particle of the present invention comprises the alloy particle preparation step of preparing by a liquid phase process an alloy particle capable of

forming a hard magnetic ordered alloy phase and the magnetic particle formation step of forming a CuAu type or Cu_3Au type magnetic particle from the prepared alloy particle.

[0039]

A method of manufacturing a magnetic particle and a magnetic particle of the present invention will be described below while describing each of the above steps. Incidentally, the magnetic particle formation step is an example of annealing treatment, which will be described below. However, the invention is not limited to this annealing treatment.

[Alloy particle preparing step]

Although an alloy particle which becomes a magnetic particle after annealing treatment can be prepared by the gaseous phase process other than the liquid phase process, the liquid phase process is desirable in consideration of the excellent mass producibility. Although conventionally known various processes can be applied as the liquid phase process, it is preferred to adopt reduction processes developed by improving the conventional liquid phase processes, and among the reduction processes, the reversed micelle process by which it is easy to control the particle size of an alloy particle is especially preferable.

[0040]

The reversed micelle process comprises the reduction step in which a reduction reaction is caused to occur by mixing at least two kinds of reversed micelle solutions and the aging step of aging at a treatment temperature after the reduction reaction.

(Reduction step)

In the reduction step, a reversed micelle solution (Solution L1), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and a reducing aqueous solution, is prepared. The reversed micelle solution (Solution L1) is hereinafter referred to simply as the solution L1.

[0041]

An oil soluble surfactant is used as the surfactant. Concretely, the sulfonic acid type (for example, a erosol OT (made by Wako Pure Chemical Industries, Ltd.), the class 4 ammonium salt type (for example, cetyl trimethylammonium bromide), the ether type (for example, pentaethylene glycol dodecyl ether), etc. can be enumerated.

[0042]

It is preferred that the amount of the surfactant in the nonaqueous organic solvent be 20 to 200 g/l.

[0043]

Alkanes, ethers, alcohols, etc. are enumerated as those which are desirable as the nonaqueous organic solvent which dissolves the surfactant. Alkanes with carbon numbers 7 to 12 are desirable as alkanes. Concretely, heptane, octane, isooctane, nonane, decane, undecane, dodecane, etc. are desirable. Diethyl ether, dipropyl ether, dibutyl ether, etc. are desirable as ethers. Ethoxyethanol, ethoxypropanol, etc. are desirable as alcohols.

[0044]

Although, compound containing alcohols, polyalcohols, H_2, HCHO, $S_2O_6^{2-}$, H_2PO_{2-}, BH_{4-}, N_2H_{5+}, H_2PO_{3-}, etc. as the reductant in the aqueous reductant solution may be use singly, it is desirable to use two kinds or more in combination. It is preferred that the amount of the reductant in the aqueous solution be 3 to 50 moles to 1 mole of metal salt.

[0045]

It is preferred that the mass ratio of the water to the surfactant in the solution L1 (water/surfactant) be not more than 20. If the mass ratio exceeds 20, this poses the problem that precipitation is apt to occur and that particles are also apt to become irregular. The mass ratio is more preferably not more than 15 and especially preferably 0.5 to 10.

[0046]

Next, apart from the above-described solution L1, a reversed micelle solution (Solutions L2), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing a plurality of kinds of metallic atoms constituting the alloy particle, are prepared. The reversed micelle solution (Solution L2) is hereinafter referred to simply as the solution L2.

[0047]

In this case, the solution L1, which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution, and a reversed micelle solution (Solution L3), which is obtained by mixing a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing one of a plurality of kinds of metallic atoms constituting the alloy particle, are prepared, the number of prepared Solutions L3 being equal to the number of the plurality of kinds of metallic atoms. The reversed micelle solution (Solution L3) is hereinafter referred to simply as the solution L3.

[0048]

The conditions (substances to be used, concentrations, etc.) for the surfactant and the nonaqueous organic solvent are the same as the solution L1. Incidentally, the same solution as the solution L1 or dissimilar solutions may be used. Furthermore, the mass ratio of the water to

the surfactant in the solution L2 are also the same as the solution L1, and the mass ratio may be the same as with the solution L1 or may be different.

[0049]

It is desirable to appropriately select the metallic salt contained in the aqueous metallic salt solution so that the magnetic particle to be prepared can form a CuAu type or Cu_3Au type hard magnetic ordered alloy.

[0050]

FeNi, FePd, FePt, CoPt, CoAu, etc. can be enumerated as CuAu type hard magnetic ordered alloys, and among others, FePd, FePt and CoPt are desirable. Ni_3Fe, $FePd_3$, Fe_3Pd, Fe_3Pt, Co_3Pt, $FePt_3$, $CoPt_3$, Ni_3Pt, $CrPt_3$ and Ni_3Mn can be enumerated as Cu_3Au type hard magnetic ordered alloys and, among others, $FePd_3$, $FePt_3$, $CoPt_3$, Fe_3Pd, Fe_3Pt and Co_3Pt are desirable.

[0051]

As concrete examples of metallic salts, it is possible to enumerate H_2PtCl_6, K_2PtCl_4, Pt $(CH_3\ COCHCOCH_3)_2$, $Na_2\ PdCl_4$, Pd $(OCOCH_3)_2$, $PdCl_2$, Pd$(CH_3\ COCHCOCH_3)_2$, $HAuCl_4$, Fe_2 $(SO_4)_3$, Fe $(NO_3)_3$, $(NH_4)_3Fe\ (C_2O_4)_3$, Fe $(CH_3\ COCHCOCH_3)_3$, $NiSO_4$, $CoCl_2$, $Co(OCOCH_3)_2$, etc.

[0052]

The concentration of the aqueous metallic salt solution (as the metallic salt concentration) is preferably 0.1 to 1000 μmol/ml and more preferably 1 to 100 μmol/ml.

[0053]

It is necessary to cause an alloy particle to undergo the transformation of the alloy phase from an unordered phase to an ordered phase by the annealing treatment which will be described later. It is preferred that at least two kinds of metallic atoms constituting the alloy particle capable of forming the CuAu type or Cu_3Au type hard magnetic ordered alloy phase be selected from the Groups VI b and VIII of the short periodic table and that at least further one kind of metallic atom be selected from the Groups I b, III a and IV a, the content of the one kind of metallic atom being 1 to 30 atom % of the whole alloy. For example, by adding one kind of metallic atom (hereinafter referred to as "a third element") selected from the Groups I b, III a and IV a, such as Sb, Pb, Bi, Cu, Ag, Zn and In, to a binary alloy constituted by two kinds of metallic atoms selected from the Groups VI b and VIII of the short periodic table, it is possible to lower the transformation temperature in causing the transformation of the alloy phase of the alloy particle from an unordered phase to an ordered phase.

[0054]

The solutions L1 and L2 prepared as described above are mixed together. In the present invention, an alloy particle which has a micro size, is excellent in monodispersibility and provides an excellent self-assembling property can be obtained by mixing together, as the plurality of solutions L1 and L2, a plurality of kinds of solutions for preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and causing the solutions to react with each other by an ultrahigh-speed stirring method in which the peripheral speed in an leading end portion of a stirring vane is not less than 5 m/second, or an in-tank mixer method in which mixing and reaction are caused to occur in a mixer provided within a reaction vessel filled with a bulk liquid and a mixed reaction solution is discharged to the bulk liquid within the reaction vessel, or a microgap mixing method in which mixing and reaction are caused to occur in a microgap formed between an inner wall of a mixer and a stirring member rotating at a high speed.

[0055]

The construction of each mixing and reaction device suitable for carrying out [1] the ultrahigh-speed stirring method, [2] the microgap mixing method and [3] the in-tank mixer method will be described below.

[1] Ultrahigh-speed stirring method

Figure 1 is a sectional view of a mixing and reaction device 10 suitable for carrying out the ultrahigh-speed stirring method.

[0056]

As shown in Figure 1, the mixing and reaction device 10 is constructed in such a manner that a high-speed stirrer 14 is provided in a vertical type mixer 12 which is formed in the shape of a cylindrical vessel. The high-speed stirrer 14 is constructed in such a manner that the upper portion of a vertical rotational shaft 16 is rotatably supported by a cover plate 18 of the mixer 12 via bearings 20 and that a motor 22, which is fixed to a seat not shown in the drawing, is connected to the top end of the rotational shaft 16. Also, two-tired upper and lower edge turbi type stirring vanes 24, 24 are installed in the bottom end portion of the rotational shaft 16 in such a manner that they are immersed in the water. It is preferred that when the diameter of the stirring vane 24 is denoted by d, the clearance between the stirring vanes 24 be in the range of 1.0 d to 0.5 d. Furthermore, it is preferred that the inside diameter D of the mixer 12 with respect to the diameter d of the stirring vane 24 be in the range of D = 1.7 d to 3.7 d. Furthermore, it is preferred that the static liquid depth is in the range of 2 d to 3 d. Incidentally, although in Figure 1, the type of the stirring vane 24 is shown as the edge turbi type, it is also possible to use the dissolver type, the paddle type, the propeller type, the flat disk type, etc. and furthermore, the

turbine type, the disk turbine type, etc. can be used. Even when the stirring vane 24 of any type is used, the high-speed stirrer 14 is constructed in such a manner that the peripheral speed at the leading end of the stirring vane 24 is preferably not less than 5 m/second and more preferably not less than 10 m/second.

[0057]

Also, a jacket 13 through which a heating medium having a relatively large heat capacity such as water and oil flows is wound around the outer circumference of the mixer 12, and a heating medium inlet port 13A and a heating medium outlet port 13B are connected to a heating medium supply device, which is not shown in the drawing. A heating medium at a temperature which can control the mixing and reaction temperature of the solutions L1, L2 within the mixer 12 to the range of -5°C to 30°C is supplied from the heating medium supply device to the jacket 13 and circulated again to the heating medium supply device. It is preferred that the mixing and reaction temperature be appropriately set within the range of -5°C to 30°C depending on the kinds of the solutions L1, L2 etc. The more preferred temperature range of the mixing and reaction temperature is 0°C to 25°C and the especially preferred temperature range thereof is 5°C to 25°C.

[0058]

Above the mixing and reaction device 10, there are provided a first preparation tank 26 which prepares the solution L1 and a second preparation tank 28 which prepares the solution L2. The first preparation tank 26 prepares the reversed micelle solution of the solution L1 by mixing a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution by use of a stirrer 30. The second preparation tank 28 prepares the reversed micelle solution of the solution L2 by mixing a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing a plurality of kinds of metallic atoms constituting the alloy particle by use of a stirrer 32. Jackets 27, 29 are provided along the outer circumferences of the first preparation tank 26 and the second preparation tank 28, respectively. When it is difficult to raise the temperature to a set mixing and reaction temperature by use of the jacket 13 of the mixer 12 alone, though this depends on the relationship between the volumes of the solutions L1, L2 which are mixed together and caused to react with each other within the mixer 12, it is possible to heat the solutions L1, L2 beforehand by use of the jackets 27, 29 of the first and second preparation tanks 26, 28.

[0059]

Addition pipes 38, 40 which have valves 34, 36, respectively, are provided in an extended condition from the bottoms of the first and second preparation tanks 26, 28 and the leading ends of the respective addition pipes 38, 40 pierce through the cover plate 18 of the mixer

12 and are caused to extend along the rotary shaft 16 of the high-speed stirrer 16 to near the solution level. In this case, although the leading ends of the addition pipes 38, 40 may be immersed in the solution, it is desirable that the leading ends of the addition pipes 38, 40 be not brought into contact with the solution surface, because it is feared that alloy particles formed by the reaction of the solutions L1, L2 within the mixer 12 may adhere to these leading ends of the addition pipes. A discharge pipe 42 of a mixed solution LM containing the alloy particles formed by a mixing reaction (an alloy-particle-containing solution) is connected to a bottom plate 12A of the mixer 12 and a valve 44 is provided in the discharge pipe 44.

[0060]

According to the mixing and reaction device 10 configured as described above, the solution L1 prepared in the first preparation tank 26 is supplied in a prescribed amount to the mixer 12 via the addition pipe 38 and the stirring vane 24 of the ultrahigh-speed stirrer 14 is caused to perform ultrahigh-speed stirring at an ultrahigh speed so that the peripheral speed at the leading end of the stirring vane 24 becomes not less than 5 m/second. Subsequently, the solution L2 from the second preparation tank 28 is added to the solution L1 within the mixer 12 via the addition pipe 40. In this case, the temperature of the solutions L1, L2 and the temperature within the mixer 12 are set at a prescribed temperature in the range of -5°C to 30°C. As a result of this, under appropriate mixing and reaction temperature conditions, in the solution L1 within the mixer 12 a very strong V-shaped spiral stream which engulfs the solution from the solution level into the interior of the solution is formed around the rotary shaft 16. Therefore, the solution L2 added to the vicinity of the rotary shaft 16 is instantaneously engulfed by this V-shaped spiral stream into the solution L1 and uniformly distributed to the whole solution L1 within the mixer 12. The mixed reaction solution LM formed by the mixing and reaction in the mixer 12 is discharged from the discharge pipe 42. The stirring time in the mixer 12, which is to be appropriately set according to the volumes of the solutions L1, L2, is preferably in the range of 1 to 30 minutes and more preferably in the range of 5 to 20 minutes. As a result of this, it is possible to prepare an alloy particle which has a particle size range of 1 to 100 nm, whose coefficient of variation in the particle size is not more than 15%, and which is excellent in the self-assembling property.

[0061]

In this case, although it is also possible to collect and store the solution L2 in the mixer 12 and to add the solution L1, in consideration of the uniformity of reduction, it is desirable to add the solution L2 to the solution L1. Although in Figure 1 the description was made by the example in which the solutions L1, L2 are mixed together and caused to react with each other, in

the case where the solution L1 and the plurality of the solutions L3 are mixed together, this method can be achieved by providing the preparation tanks and addition pipes in numbers equal to the number of the solutions.

[0062]

Incidentally, the mixing and reaction device 10 for carrying out the ultrahigh-speed stirring method is not limited to the mixing and reaction device of Figure 1, and any mixing and reaction device can be used so long as the mixing and reaction device is constructed in such a manner that it can supply a plurality of kinds of solutions for preparing an alloy particle to the interior of the mixer 12 and cause the mixing and reaction to occur by the liquid phase process and at the same time it can ensure that the peripheral speed at the leading end of the stirring vane 24 is not less than 5 m/sec.

[2] Microgap mixing method

Figure 2 is a sectional view of a mixing and reaction device 50 suitable for carrying out the microgap mixing method.

[0063]

As shown in Figure 2, the mixing and reaction device 50 is constructed in such a manner that in a vertical type mixer 52 which is formed in the shape of a cylindrical vessel whose top and bottom are hermetically closed, there is provided a column-shaped rotary drum 54 having a diameter a little smaller than the inside diameter of this mixer 52. A rotary shaft 56 which is perpendicular along the center of rotation S is provided in the rotary drum 54, and the upper and lower portions of the rotary shaft 56 are rotatably supported by the top plate 52A and bottom plate 52B of the mixer 52 via bearings 58, 58. A motor 60, which is fixed to a seat not shown, is connected the top end of the rotary shaft 56.

[0064]

As shown in Figure 2 and Figure 3 (a cross-sectional view of the mixer 52), an annular microgap 62 (a microgap) is formed between the circumferential surface of the inner wall of the mixer 52 and the outer circumferential surface of the rotary drum 54. When the distance D_1 from the center of rotation S of the rotary drum 54 to the leading end thereof is put as 1, this microgap 62 is formed by setting the distance D_2 to the inner wall of the mixer 52 having the shortest distance from the center of rotation S of the rotary drum 54 in the range of 1.001 to 1.200.

[0065]

Near the mixing and reaction device 10, there are provided a first preparation tank 64 which prepares the solution L1 and a second preparation tank 66 which prepares the solution L2.

The first preparation tank 64 prepares the reversed micelle solution of the solution L1 by mixing a nonaqueous organic solvent containing a surfactant and an aqueous reductant solution by use of a stirrer 68. The second preparation tank 66 prepares the reversed micelle solution of the solution L2 by mixing a nonaqueous organic solvent containing a surfactant and an aqueous metallic salt solution containing a plurality of kinds of metallic atoms constituting the alloy particle by use of a stirrer 70.

[0066]

Supply pipes 80, 82 which have valves 72, 74, and supply pumps 76, 78, respectively, are provided in an extended condition from the bottoms of the first and second preparation tanks 64, 66 and the leading ends of the respective solution supply pipes 80, 82 are connected to supply ports 84, 86 which are formed in opposed positions of the side surface of the bottom end portion of the mixer 52. On the side surface of the top end portion of the mixer 52 is formed a discharge port 88 of the mixed reaction solution LM and the discharge tube 90 is connected to the discharge port 88.

[0067]

Jackets 51, 69, 71, 81, 83 through which a heating medium having a relatively large heat capacity such as water and oil flows are wound around the respective outer circumferences of the mixer 52, first and second preparation tanks 64, 66, and supply pipes 80, 82, and the jackets 51, 69, 71, 81, 83 are connected to a heating medium supply device, which is not shown in the drawing. A heating medium at a temperature which can control the mixing and reaction temperature of the solutions L1, L2 within the mixer 12 to the range of -5°C to 30°C is supplied from the heating medium supply device to the jacket 51, 69, 71, 81, 83 and circulated again to the heating medium supply device. It is preferred that the mixing and reaction temperature be appropriately set within the range of -5°C to 30°C depending on the kinds of the solutions L1, L2 etc. The more preferred temperature range of the mixing and reaction temperature is 0°C to 25°C and the especially preferred temperature range thereof is 5°C to 25°C.

[0068]

According to the mixing and reaction device 50 configured as described above, the solution L1 and the solution L2, which have been prepared in the first preparation tank 64 and the second preparation tank 66, respectively, under appropriate mixing and reaction temperature conditions, with the rotary drum 54 kept rotated at an ultrahigh speed so that the peripheral speed at the leading end of the rotary drum 54 (in a position of the circumferential surface at the leading end), are supplied by the solution supply pumps 76, 78 to the inside of the microgap 62 of the mixer 52. While being subjected to a uniform shearing force by the circumferential surface of

the inner wall of the fixed mixer 52 and the outer circumferential surface of the rotary drum which is rotating, the solutions L1, l2 fed into the microgap 62 move in a spiral form within the microgap 62 from the bottom end to the top end of the mixer 52 and reaches the discharge port 88 and discharged from the discharge tube 90. As a result of this, it is possible to ensure that the solutions L1, L2 are instantaneously and efficiently mixed together and caused to react with each other in the microgap 62, an alloy particle which has a micro size and is excellent in monodispersibility is formed. As a result of this, it is possible to prepare an alloy particle which has a particle size range of 1 to 100 nm, whose coefficient of variation in the particle size is not more than 15%, and which is excellent in the self-assembling property. In this case, if the above-described distance D_2 is less than 1.001 and too short, the shearing force becomes nonuniform due to the effect of the surface roughness of the circumferential surface of the inner wall of the mixer 52 and the outer circumferential surface of the rotary drum 54, with the result that the monodispersibility of a prepared alloy particle becomes apt to worsen. If the distance D_2 exceeds 1.200 and is too large, the shearing force contributing to the solutions L1, L2 decreases, with the result that the size of a prepared alloy particle becomes large and that at the same time the monodispersibility also become apt to worsen.

[0069]

Figure 4 is an example of a modification of a mixing and reaction device 100 for carrying out the microgap mixing method. Descriptions are given by assigning like reference numerals to the same members as in the mixing and reaction device 50 of Figure 2.

[0070]

As shown in Figure 4, the mixing and reaction device 100 is constructed in such a manner that the diameter of an inner wall of a mixer 52 with respect to the outside diameter of a rotary drum 54 decreases from the bottom end to the top end of the mixer 52 and a microgap 62 becomes narrow from the bottom end to the top end of the mixer 52. According to this mixing and reaction device 100, the flow velocity of the solutions L1, L2 supplied into the microgap 62 becomes high as solutions L1, L2 move from the bottom end to the top end of the mixer 52, with the result that it is possible to gradually increase the shearing force given to the solutions L1, L2. As a result of this, it is possible to obtain an alloy particle which has a micro size, is excellent in monodispersibility and provides an especially excellent self-assembling property. In this case, the distance D_1 from the center of rotation S of the rotary drum 54 to the leading end thereof and the distance D_2 to the inner wall of the mixer 52 having the shortest distance from the center of rotation S of the rotary drum 54 are the sizes in the top end position of the mixer 52, as shown in Figure 4.

[0071]

Figure 5 is another example of a modification of a mixing and reaction device 110 for carrying out the microgap mixing method. Descriptions are given by assigning like reference numerals to the same members as in the mixing and reaction device 50 of Figure 2.

[0072]

As shown in Figure 5, in the mixing and reaction device 110, in place of the rotary drum 54 a plurality of flat disks 92, 92 ... are radially arranged in prescribed locations at a prescribed gap from the rotary shaft 56. In this case, it is preferred that the distance between the flat disks 92 be equal to the microgap 62 formed between the outer circumferential surface of the flat disk 92 and the circumferential surface of the inner wall of the mixer 52. Also in the mixing and reaction device 110 of Figure 5, it is possible to obtain an alloy particle which has a micro size, is excellent in monodispersibility and provides an especially excellent self-assembling property.

[0073]

Incidentally, the mixing and reaction device for carrying out the microgap mixing method is not limited to those of Figure 2, Figure 4 and Figure 5. It is possible to use any mixing and reaction device which has the microgap 62 formed between the inner wall of the mixer 52 and the stirring member 54, 92 which rotates at a high speed, and in which by using a device in which when the distance from the center of rotation S of the stirring member 54, 92 to the leading end thereof is put as 1, the distance to the inner wall having the shortest distance from the center of rotation S of the stirring member 54, 92 is set in the range of 1.001 to 1.200, the solutions L1, L2 are supplied to the microgap 62 and mixed together and caused to react with each other and at the same time the mixed reaction solution LM is discharged from the microgap 62.

[3] In-tank mixer method

Figure 6 and Figure 7 are each a sectional view of a mixing and reaction device 120 suitable for carrying out the in-tank mixer method.

[0074]

As shown in these figures, the mixing and reaction device 120 is constructed in such a manner that a mixer 126 whose top end portion is open and a circular opening 124 for circulation is formed in the bottom end portion is provided within a reaction vessel 122 filled with a bulk liquid and the interior of the mixer 126 is also filled with a bulk liquid. The above-described nonaqueous organic solvent containing a surfactant is used as the bulk liquid. A jacket 123 through which a heating medium having a relatively large heat capacity such as water and oil flows is wound around the outer circumference of the reaction vessel 122, and the jacket 123 is

connected to a heating medium supply device, which is not shown in the drawings. A heating medium at a temperature which can control the mixing and reaction temperature of the solutions L1, L2 within the reaction vessel 122 to the range of -5°C to 30°C is supplied from the heating medium supply device to the jacket 123 and circulated again to the heating medium supply device. It is preferred that the mixing and reaction temperature be appropriately set within the range of -5°C to 30°C depending on the kinds of the solutions L1, L2 etc. The more preferred temperature range of the mixing and reaction temperature is 0°C to 25°C and the especially preferred temperature range thereof is 5°C to 25°C.

[0075]

A pair of addition pipes 128, 130 for adding the solutions L1, L2, which has valves 125, 127, is provided in an extended condition so that the pair of addition pipes 128, 130 passes through the reaction vessel 122 from the outside of the reaction vessel 122, enters drilled passages formed in a bottom plate of the mixer 126 and reaches the edge of an opening for circulation 124. The addition ports of the addition pipes 128, 130 of the solutions L1, l2 are disposed in the opening for circulation 124 in directions which are opposite to each other. In the vicinity of the opening for circulation 124 within the mixer 126, tow-tired upper and lower stirring vanes 134, 134 which are supported by a rotary shaft 132 are provided, and the rotary shaft 132 is rotated by a motor 138. Among these two stirring vanes 134, 136, the lower stirring vane 136 is formed so as to be able to cause rapid mixing and reaction of the solutions L1, L2. Therefore, it is preferred that the peripheral speed at the leading end of the lower stirring vane 136 be not less than 5 m/second. On the other hand, the upper stirring vane 134 is formed so as to be able to generate a circulating current which flows from the open top end portion of the mixer 126 to the reaction vessel 122 and returns from the opening for circulation 124 to the mixer 126. The circulating flow rate of the circulating current generated by the upper stirring vane 134 is designed so that it becomes not less than 500 l/minute in the position of the opening for circulation 124. An increase in the circulating flow rate can be achieved by increasing the vane diameter of the upper stirring vane 134, increasing the opening diameter of the opening for circulation 124, etc.

[0076]

The added flow rates of the solutions L1, l2 added from the addition pipes 128, 130 are designed so that not less than 20 l/minutes, preferably not less than 30 l/minutes, and especially preferably not less than 40 l/minutes can be controlled with a good accuracy. An increase in the added flow rate can be achieved by increasing the pipe diameter of the addition pipes 128, 130 and by generating the above-described circulating current of a large flow velocity near the

opening for circulation 124 to which the solutions L1, L2 are added thereby to generate a suction force near the addition ports of the addition pipes 128, 130. Furthermore, an increase in the control accuracy of the added flow rate can be achieved by installing a flow regulating valve 140 of the construction shown in Figure 8 in each of the addition pipes 128, 130.

[0077]

As shown in Figure 8, the valve main body 142 of the flow regulating valve 140 is constituted by a valve casing 144 and a valve plate 146, the interior of the valve main body 142 is provided with a valve chest 150 having an inlet chamber 148, and the solution L1 or the solution L2 flows in from an inlet port 152. The valve chest 150 is provided with an outlet port 154 of openings 154a, 154b in the direction orthogonal to the outflow direction of the fluid. The outlet port 154 is provided with a valve rod 156 which is moved in a sliding manner using a motor (not shown) as the drive source. The opening area of the outlet port 154 which is exposed to the valve chest 150 increases or decreases proportionally according to the sliding movement distance of this valve rod 156, and the solution L1 or the solution L2 which has flown into the inlet chamber 148 of the valve chest 150 is caused to flow out of the outlet port 154 at a flow rate proportional to the opening area. This flow regulating valve 140 provides a good linearity between the valve opening and the flow rate and can perform flow rate control with good accuracy in a wide flow rate range.

[0078]

According to the mixing and reaction device 120 configured as described above, the solutions L1, L2 added from the addition pipes 128, 130 flow into the mixer under appropriate mixing and reaction temperature conditions 126 while being diluted by the bulk liquid circulated by the opening for circulation 124 and are mixed together and caused to react with each other within the mixer 126. And the mixed reaction solution LM is discharged by the circulating bulk liquid from the mixer into the reaction vessel 122. In this manner, by performing mixing and reaction with the solutions L1, L2 and the mixed reaction solution LM kept diluted with the bulk liquid, the opportunity of contact of generated alloy particles is reduced and, therefore, the growth of particles can be suppressed. Furthermore, by ensuring a circulating flow rate of the bulk liquid of not less than 500 l/minutes in the position of the opening for circulation 124, the solutions L1, L2 added from the addition pipes 128, 130 can be immediately diluted with the bulk liquid and by ensuring an added flow rate of the solutions L1, l2 of not less than 20 l/minute, the reaction can be completed in a short time, with the result that an alloy particle which has a micro size and is excellent in monodispersibility is formed. As a result of this, it is possible to prepare

an alloy particle which has a particle size range of 1 to 100 nm, whose coefficient of variation in the particle size is not more than 15%, and which is excellent in the self-assembling property.

[0079]

Incidentally, the mixing and reaction device 120 for carrying out the in-tank mixer method is not limited to the above-described configurations. It is possible to use any mixing and reaction device in which, by using a device in which there is provided the reaction vessel 122 which is filled with a bulk liquid and provided therein with the mixer 126 which has the stirring vane 136 rotating at a high speed and is provided with the opening 124 through which the bulk liquid is circulated to and from the interior of the reaction vessel 122, the solutions L1, L2 for preparing the alloy particle are supplied to the interior of the mixer 126, where the solutions are mixed together and caused to react with each other by a liquid phase process, and at the same time the mixed reaction solution LM can is discharged from the mixer 126 to the reaction vessel 122 by a circulating stream of the bulk liquid.

[0080]

By mixing the solution L1 and the solution L2 by use of a mixing and reaction device for carrying out the [1] ultrahigh-speed stirring method, [2] microgap mixing method and [3] in-tank mixer method described above, it is possible to prepare an alloy particle which meets all of the requirements for a micro size, monodispersibility and transformation facilitativeness in the alloy particle preparation step.

[0081]

It is preferred that the mixing and reaction temperature of the reduction reaction by the above-described mixing be a constant temperature in the range of -5°C to 30°C. If the mixing and reaction temperature is less than -5°C, this poses the problem that a water phase condenses, making the reduction reaction nonuniform. If the mixing and reaction temperature exceeds 30°C, coalescence and precipitation tend to occur and the system may sometimes become unstable. The reduction temperature is preferably in the range of 0°C to 25°C and more preferably in the range of 5°C to 25°C. "A constant temperature" here device that when a set temperature is T (°C), this T is in the range of T ±3°C. Incidentally, also in this case, the upper limit and lower limit to this T are the above-described -5°C to 30°C. The reduction reaction time, which must be appropriately selected according to the reversed micelle capacity etc., is preferably 1 to 30 minutes and more preferably 5 to 20 minutes.

[0082]

In the above-described reduction step, it is preferred that to at lest either of the solutions L1 and L2, at least one kind of dispersant which contains 1 to 3 amino groups or carboxyl groups

be added in an amount of 0.001 to 10 moles per mole of alloy particle to be prepared. By adding such a dispersant, it is possible to obtain an alloy particle which is more monodispersible and free from coalescence. When the amount of added dispersant is less than 0.001 mole, it may be sometimes impossible to further improve the monodispersibility of an alloy particle. When the amount of added dispersant exceeds 10 mole, coalescence may sometimes occur.

[0083]

An organic compound having a group which is adsorbed on the surface of an alloy particle is desirable as a dispersant. Concretely, dispersants which have 1 to 3 amino groups, carboxyl groups, sulfonate groups or sulfinate groups are preferred and these may be used singly or in combination.

[0084]

These compounds have the following structural formulas: R-NH_2, NH_2-R-NH_2, NH_2-R (NH_2)-NH_2, R-COOH, COOH-R-COOH, COOH-R (COOH)-COOH, R-SO_3H, SO_3H-R-SO_3H, SO_3H-R (SO_3H)- SO_3H, R-SO_2H, SO_2H-R-SO_2H, SO_2H-R (SO_2H)-SO_2H. The R in the formulas each denotes a linear, branched or cyclic saturated or unsaturated hydrocarbon.

[0085]

A specially desirable compound as a dispersant is oleic acid. Oleic acid, which is a surfactant which is widely known in the stabilization of colloids, has been used in protecting metallic particles of iron etc. The relatively long chain of oleic acid provides a cubic obstacle important for counteracting the strong magnetic interaction between particles (for example, oleic acid has 18 carbon chains and its length is up to 20 angstroms (up to 2 nm). Oleic acid is not a fatty acid and has one double bond).

[0086]

As with oleic acid, similar long-chain carboxylic acids, such as erucic acid and linoleic acid, are also used (for example, long-chain organic acids having 8 to 20 carbon atoms can be used singly or in combination). Oleic acid (for example, olive oil) is desirable because it is an inexpensive natural resource capable of being easily obtained. Furthermore, oleylamine derived from oleic acid is also a dispersant which is as useful as oleic acid.

[0087]

It might be thought that in the reduction step as described above, metals which are base in terms of redox potentials (metals of not more than -0.2 V (vs. NHE) or so), such as Co, Fe, Ni and Cr, are reduced in a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and precipitate in a state of micro size and monodispersion. It might be thought that after that, in the temperature rise step and the aging step which will be described later, with the precipitated base

metals serving as nuclei, metals which are noble in terms of redox potentials (metals of not less than -0.2 V (vs. NHE) or so), such as Pt, Pd and Rh, are reduced on the surface, displaced and precipitate by the base metal. It might be thought that the ionized base metals are reduced again by a reductant and precipitate. An alloy particle capable of forming CuAu type or Cu_3Au type hard magnetic ordered alloy is obtained by repetitions of such reactions.

[0088]

Next, a description will be given of the aging step which raises, after the completion of the reduction step, the mixing and reaction solution to an aging temperature higher than the mixing and reaction temperature of -5°C to 30°C in the reduction step as described above.

(Aging step)

It is preferred that the aging temperature be a constant temperature in the range of 30 to 90°C and, this temperature should be higher than the temperature of reduction reaction. It is preferred that the aging time be 5 to 180 minutes. If the aging temperature and aging time shift to the high temperature and long time side from above range, coalescence or precipitation is apt to occur. Conversely, if the aging temperature and aging time shift to the low temperature and short time side, the reaction may not sometimes complete, resulting in a change in the composition. The aging temperature and aging time are preferably 40 to 80°C and 10 to 150 minutes and more preferably 40 to 70°C and 20 to 120 minutes.

[0089]

"A constant temperature" here is synonymous with the case of the temperature of reduction reaction (however, in this case, "the reduction temperature" becomes "the aging temperature"). In particular, however, "a constant temperature" is preferably higher than the temperature of reduction reaction by not less than 5°C within the above-described range of the aging temperature (30 to 90°C) and more preferably higher than the temperature of reduction reaction by not less than 10°C. In the case of less than 5°C, a prescribed composition may not sometimes be obtained.

[0090]

In the aging step as described above, noble metals precipitate on the base metals which were reduced and precipitated in the reduction step. That is, because the reduction of noble metals occurs only on base metals and base metals and noble metals dot not separately precipitate, it is possible to prepare an alloy particle capable of efficiently forming a CuAu type or Cu_3Au type hard magnetic ordered alloy at a high yield and according to a prescribed composition and it is possible to control the alloy particle to a desired composition. Furthermore, by appropriately

adjusting the temperature and the stirring rate of during aging, it is possible to obtain a desired particle diameter of the obtained alloy particle.

[0091]

It is desirable to provide the cleaning and dispersion step of cleaning the solution after aging with a mixed solution of water and first class alcohol, then performing precipitation treatment with first class alcohol thereby to generate precipitates, and dispersing the precipitates with an organic solvent.

[0092]

Impurities are removed by providing this cleaning and dispersion step and it becomes possible to further improve applicability when the magnetic layer of a magnetic recording medium is formed by application. Cleaning and dispersion are each performed at least once and preferably each twice or more.

[0093]

Although first class alcohol used in cleaning is not especially limited, methanol, ethanol, etc. are desirable. The volume mixing ratio (water/first class alcohol) is preferably in the range of 10/1 to 2/1 and more preferably in the range of 5/1 to 3/1. If the ratio of water is high, the surfactant may not sometimes be easily removed. Conversely, if the ratio of first class alcohol is high, coalescence may sometimes occur.

[0094]

Alloy particles dispersed in a solution (an alloy-particle-containing solution) are obtained in a manner as described above.

[0095]

The alloy articles are monodispersed. Therefore, even when the alloy articles are applied to a backing, these particles do not coalesce together and can be kept in a uniformly dispersed state. Therefore, even when annealing treatment is performed, the respective alloy particles do not coalesce together and hence it is possible to ensure efficient hard magnetizing, resulting in excellent applicability. Furthermore, the alloy particle is excellent in the self-assembling property because it is prepared by the above-described high-pressure mixing methods, and annealing treatment enables the alloy particle to undergo transformation from an unordered phase to an ordered phase easily and positively. This permits efficient hard magnetizing.

[0096]

From the standpoint of lowering the noise level, it is desirable that the particle size of an alloy particle before oxidation treatment, which will be described later, be small. However, if

the particle size is too small, supermagnetism occurs after annealing and the particle may sometimes become unsuitable for magnetic recording. In general, the particle size is preferably in the range of 1 to 100 nm, more preferably in the range of 3 to 20 nm, and most preferably in the range of 3 to 10 nm.

(Reduction process)

A general reduction process for preparing alloy particles is described here.

[0097]

Although there are various methods of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy in the reduction process, it is desirable to apply a method of reducing at least a metal which is base in terms of redox potential (hereinafter may sometimes be referred to simply as "a base metal") and a metal which is noble in terms of redox potential (hereinafter may sometimes be referred to simply as "a noble metal") by use of a reductant etc. in an organic solvent or water or a mixed solution of an organic solvent and water. The order of reduction of the base metal and noble metal is not especially limited and the base metal and noble metal may be simultaneously reduced.

[0098]

Alcohols, polyalcohols, etc. can be used as the above-described organic agent. Methanol, ethanol, butanol, etc. can be enumerated as alcohols and ethylene glycerin, glycerol, etc. can be enumerated as polyalcohols.

[0099]

Incidentally, examples of a CuAu type or Cu_3Au type ferromagnetic ordered alloy are the same as in the case of the above-described reversed micelle process.

[0100]

The methods described in the paragraphs 18 to 30 etc. of the Japanese Patent Application No. 2001-269255 can be applied as a method of preparing an alloy particle by precipitating a noble metal beforehand.

[0101]

Pt, Pd, Rh, etc. can be advantageously used as metals which are noble in terms of redox potential and $H_2\,PtCl_6 \cdot 6H_2O$, Pt $(CH_3\,COCHCOCH_3)_2$, $RhCl_3 \cdot 3H_2O$, Pd $(OCOCH_3)_2$, $PdCl_2$, Pd $(CH_3\,COCHCOCH_3)_2$, etc. can be used by being dissolving in a solvent. The concentration of a metal in the solution is preferably in the range of 0.1 to 1000 μmol/ml and more preferably in the range of 0.1 to 100 μmol/ml.

[0102]

Co, Fe, Ni and Cr can be favorably used as metals which are base in terms of redox potential and Fe and Co are especially preferably used. Au such metals, $FeSO_4 \cdot 7H_2O$, $NiSO_4 \cdot 7H_2O$, $CoCl_2 \cdot 6H_2O$, Co $(OCOCH_3)_2 \cdot 4H_2O$, etc. can be used by being dissolved in a solvent. The concentration of a metal in the solution is preferably in the range of 0.1 to 1000 μmol/ml and more preferably in the range of 0.1 to 100 μmol/ml.

[0103]

As with the above-described reversed micelle process, it is desirable to lower the temperature of transformation to a hard magnetic ordered alloy by adding a third element to a binary alloy. The amount of an added metal is the same as with the reversed micelle process.

[0104]

For example, in a case where a base metal and a noble metal are reduced in this order and caused to precipitate, it is desirable to first reduce the base metal or the base metal and part of the noble metal by use of a reductant having a more base reduction potential than -0.2 V (vs. NHE), add the reduced metals to the source of the noble metal, then perform reduction by use of a reductant which is nobler in terms of redox potential than -0.2 V (vs. NHE) and perform reduction by use of a reductant having a reduction potential which is more base than -0.2 V (vs. NHE).

[0105]

Redox potentials depend on the pH of the system. Alcohols, such as 1,2-hexadecandiol, glycerins, H_2 and HCHO are advantageously used as reductants which are nobler than -0.2 V (vs. NHE).

[0106]

As reductants which are more base than -0.2 V (vs. NHE), $S_2O_6^{2-}$, H_2PO_{2-}, BH_{4-}, N_2H_{5+}, H_2PO_{3-} can be advantageously used.

[0107]

Incidentally, when a zero-valent metallic compound, such as Fe carbonyl, is used as the material for a base metal, it is unnecessary to use a reductant for the base metal

[0108]

By causing an adsorbent to be present during the reduction and precipitation of a noble metal, it is possible to prepare an alloy particle in a stable manner. It is desirable to use a polymer and a surfactant as an adsorbent.

[0109]

Polyvinyl alcohol (PVA), poly-N-vinyl-2-pyrrolidone (PVP), gelatin, etc. can be enumerated as the above-described polymer. Among others, PVP is particularly preferable.

[0110]

The molecular weight is preferably in the range of 20000 to 60000 and more preferably in the range of 30000 to 50000. The quantity of a polymer is preferably in the range of 0.1 to 10 times the mass of a generated alloy particle and more preferably in the range of 0.1 to 5 times.

[0111]

It is preferred that a surfactant which is advantageously used as an adsorbent contain "an organic stabilizer" which is a long-chain organic compound expressed by the general formula R-X. The R in the above general formula denotes "a tail group" which is a straight-chain or branched hydrocarbon or fluorocarbon chain and usually contains 8 to 22 carbon atoms. The X in the above general equation denotes "a head group" which is a portion (X) that supplies a specific chemical bond to the alloy particle surface, and it is preferred that X be any one of sulfinate (-SOOH), sulfonate ($-SO_2OH$), phosphinate (-POOH), phosphonate ($-OPO(OH)_2$), carboxylate and thiol.

[0112]

It is preferred that the above-described organic stabilizer be any one of sulfonic acid ($R-SO_2OH$), sulfinic acid (R-SOOH), phosphinic acid (R_2POOH), phosphonic acid ($R-OPO(OH)_2$), carboxylic acid (R-COOH), thiol (R-SH), etc. As with the reversed micelle process, however, the use of oleic acid is especially preferred than any other above-enumerated substances.

[0113]

Combinations of the above-described phosphine and an organic stabilizer (triorganophosphine/acid etc.) can provide excellent controllability for the growth and stabilization of particles. Although didecyl ether and didodecyl ether can also be used, phenyl ether or n-octyl ether is advantageously used as a solvent for its low cost and high boiling point.

[0114]

However, a general reduction process for preparing alloy particles is performed at high temperatures compared to the mixing and reaction temperature in the range of -5°C to 30°C in the mixing and reaction using the high-speed stirring methods as in the present invention. That is, it is general practice to cause the reaction to occur at a temperature in the range of 80°C to 360°C owing to necessary alloy particles and the boiling temperature of a solvent and the temperature range of 80°C to 240°C is more preferred. In the case of a general reduction process, particles may sometimes not grow when the reaction temperature is lower than this temperature range. On the other hand, if the temperature is higher than this range, particles grow without being controlled and the generation of undesirable by-products may sometimes increase.

[0115]

As with the reversed micelle process, the particle diameter of an alloy particle is preferably in the range of 1 to 100 nm, more preferably in the range of 3 to 20 nm, and further more preferably in the range of 3 to 10 nm, in the same manner as in the present invention.

[0116]

The seed crystal process is effective as a method of increasing the particle size (particle diameter). In order to use alloy particles as a magnetic recording medium, filling alloy particles at fine densities is preferable for increasing the recording capacity and, for this purpose, the standard deviation of an alloy particle size is preferably below 10% and more preferably not more than 5%. In the present invention, the size of an alloy particle is specified by the coefficient of variation, and the coefficient of variation is not more than 15% and preferably not more than 10%.

[0117]

If the particle size is too small, superparamagnetism occurs and this is undesirable. Therefore, in order to increase the particle size, it is desirable to adopt the seed crystal process as described above. On this occasion, there is a case where a metal which nobler than the metal which constitutes particles is caused to precipitate. Because the oxidation of particles is feared in this case, it is desirable that the particles be subjected to hydrogenation treatment beforehand.

[0118]

Although it is desirable that the outermost layer of an alloy particle be constituted by a noble metal from the standpoint of the prevention of oxidation, such a noble metal is apt to coalesce and hence in the present invention, it is desirable that the outermost layer of an alloy particle be constituted by an alloy of a noble metal and a base metal. According to the liquid phase process as described above, such a constitution can be easily and efficiently realized.

[0119]

Removing salts from the solution after the preparation of alloy particles is desirable from the standpoint of improving the dispersion stability of alloy particles. In removing salts, there is a method by which alcohol is excessively added thereby to cause slight coalescence and the salts are removed together with a supernatant by causing natural sedimentation or centrifugal sedimentation. Because coalescence is apt to occur in this method, it is desirable to adopt the ultrafiltration method.

[0120]

Alloy particles dispersed in a solution (an alloy-particle-containing solution) can be obtained as described above.

[0121]

A transmission electron microscope (TEM) can be used in evaluating the particle size of an alloy particle. Although electron diffraction by a TEM may be used in determining the crystal system of an alloy particle or a magnetic particle, the use of X-ray diffraction is desirable because of high accuracy. In the composition analysis of the interior of an alloy particle or a magnetic particle, it is desirable to perform an evaluation by adding EDAX to FE-TEM capable of reducing the section of electron beams. Furthermore, the evaluation of the magnetic properties of an alloy particle or a magnetic particle can be performed by use of VSM.

[Oxidation treatment step]

By subjecting a prepared alloy particle to oxidation treatment, it is possible to efficiently manufacture a magnetic particle having hard magnetism without raising the temperature during annealing treatment, which is performed later in a nonoxidizing atmosphere. It might be thought that this is due to the phenomenon which will be described below.

[0122]

That is, first, by oxidizing an alloy particle, oxygen enters the crystal lattice of the alloy particle. When annealing treatment is performed, with oxygen in the crystal lattice, the oxygen is released from the crystal lattice due to heat. The liberation of the oxygen causes a defect. Because the migration of metallic atoms constituting the alloy becomes easy through this defect, phase transformation becomes apt occur even at a relatively low temperature. Therefore, by subjecting an alloy particle having a good self-assembling property prepared by the above-described high-pressure mixing method to oxidation treatment, it becomes easier to lower the temperature of annealing treatment.

[0123]

This phenomenon can be supposed, for example, by measuring the EXAFS (extended X-ray absorption fine structure) of an alloy particle after oxidation treatment and an annealed magnetic particle.

[0124]

For example, in an Fe-Pt alloy particle not subjected to oxidation treatment, the presence of an Fe atom and the bond between a Pt atom and an Fe atom can be recognized.

[0125]

In contrast to this, in an alloy particle subjected to oxidation treatment, the presence of the bond between an Fe atom and an oxygen atom can be recognized. However, the bond between a Pt atom and an Fe atom is scarcely seen. This device that the Fe-Pt bond and the Fe-Fe bond have been cut by oxygen atoms. It might be thought that this is the reason why a Pt atom and an Fe atom can migrate easily during annealing.

[0126]

And after this alloy particle is subjected to annealing treatment, the presence of oxygen cannot be recognized and the presence of the bond to a Pt atom or an Fe atom can be recognized around an Fe atom.

[0127]

In view of the above phenomenon, it will be understood that phase transformation does not proceed easily unless oxidation is performed and that it becomes necessary to raise the temperature of annealing treatment. However, it might also be though that if excessive oxidation is performed, the interaction between a metal which is easily oxidized, such as Fe, and oxygen becomes too strong, with the result that a metal oxide is formed.

[0128]

Therefore, it becomes important to control the oxidation state of an alloy particle and for this purpose, it is necessary to set optimum oxidation treatment conditions.

[0129]

In oxidation treatment, for example, in a case where alloy particles are prepared by the above-described liquid phase process, it is necessary only that a gas containing at least oxygen be supplied to an alloy-particle-containing solution after the preparation of the alloy particles.

[0130]

The partial pressure of oxygen in this case is preferably in the range of 10 to 100% of the total pressure and more preferably in the range of 15 to 50%. The temperature of oxidation treatment is preferably in the range of 0 to 100°C and more preferably in the range of 15 to 80°C.

[0131]

It is preferred that the oxidation state of alloy particles be evaluated by EXAFS etc. and in consideration of that an Fe-Fe bond and a Pi-Fe bond are cut by oxygen, the number of bonds between base metals such as Fe and oxygen is preferably in the range of 0.5 to 4 and more preferably in the range of 1 to 3.

[Annealing treatment step]

An alloy particle subjected to oxidation treatment forms an unordered phase. As described above, hard magnetism cannot be obtained from an unordered phase. In order to form an ordered phase, therefore, it is necessary to perform heat treatment (annealing). In this heat treatment, it is necessary to use differential thermal analysis (DTA) to determine the transformation temperature at which the alloys constituting an alloy particle undergoes transformation between an ordered phase and an unordered phase and perform the heat treatment at a temperature of not less than this temperature.

[0132]

Although the above-described transformation temperature is usually about 500°C, it is possible to use a temperature below the usual temperature, because the self-assembling property of the prepared alloy particle is improved by the mixing by the high-pressure mixing methods in the above-described reduction step. Therefore, the temperature of annealing treatment is preferably not less than 100°C and more preferably in the range of 100 to 500°C. Furthermore, the temperature may sometimes be fallen by the addition of a third element.

[0133]

When annealing treatment is performed in the state of a particle, the migration of particles is apt to occur and coalescence is apt to occur. For this reason, although a high coercive force is obtained, this tends to pose the problem that the particle size increases. From the standpoint of the prevention of the coalescence of alloy particles, it is preferred that alloy particles applied to a backing etc. be subjected to annealing treatment.

[0134]

Furthermore, by annealing alloy particles on a backing to form magnetic particles, it is possible to use a layer constituted by such magnetic particles to be a magnetic layer as a magnetic recording medium.

[0135]

Any backing, whether it is made of an inorganic substance or an organic substance, may be used so long as it is a backing used in a magnetic recording medium.

[0136]

As the backings of an inorganic substance, Al, Mg-containing alloys such as Al-Mg and Mg-Al-LMn, glass, quartz, carbon, silicon, ceramics, etc. are used. These backings are excellent in impact resistance and has rigidity suitable for thin thickness design and high speed rotation. These backings have the characteristic that of high heat resistance as organic substance.

[0137]

As the backings of an organic substance, polyesters such as polyethylene terephthalate and polyethylene naphthalate, polyolefins, cellulose triacetate, polycarbonate, polyamides (including fatty polyamide and aromatic polyamides such as aramid), polyimide, polyamide-imide, polysulfone, polybenzoxazole, etc. can be used.

[0138]

In applying alloy particles to a backing, it is necessary only that various additives be added to the above-described alloy-particle-containing solution subjected to oxidation treatment as required and then alloy particles be applied to the backing.

[0139]

It is desirable that the content of alloy particles in this case be a required concentration (0.01 to 0.1 mg/ml).

[0140]

Air doctor coating, blade coating, rod coating, extrusion coating, air knife coating, squeeze coating, impregnation coating, reverse roll coating, transfer roll coating, offset gravure coating, kiss coating, cast coating, spray coating, spin coating, etc. can be used as methods of application to a backing.

[0141]

As an atmosphere during annealing treatment, a nonoxidizing atmosphere of H_2, N_2, Ar, He, Ne, etc. should be used in order to ensure that phase transformation proceeds efficiently thereby to prevent the oxidation of alloys.

[0142]

Particularly, from the standpoint of causing the oxygen to remove present on lattices by oxidation treatment, it is desirable to use a reducing atmosphere of methane, ethane, H_2, etc. Furthermore, from the standpoint of keeping the particle diameter, it is desirable to perform annealing treatment in a magnetic field in a reducing atmosphere. Incidentally, when an H_2 atmosphere is used, it is desirable that an inert gas be mixed from the standpoint of explosion protection.

[0143]

In order to prevent the coalescence of particles during annealing, it is desirable first to perform annealing treatment at a temperature of not more than the transformation temperature in an inert gas thereby carbonize a dispersant and then to perform annealing treatment at a temperature of not less than the transformation temperature in a reducing atmosphere. At this time, the most desirable mode is first to perform the above-described annealing treatment at a temperature of not more than the transformation temperature as required, then to apply an Si-base resin etc. to a layer constituted by alloy particles, and lastly to perform annealing treatment at a temperature of not less than the transformation temperature.

[0144]

By performing annealing treatment as described above, it is ensured that alloy particles undergo phase transformation from an unordered phase to an ordered phase and magnetic particles having hard magnetism can be obtained.

[0145]

The coercive force of a magnetic particle manufactured by the above-described method of manufacturing a magnetic particle according to the present invention is preferably in the range of 95.5 to 955 kA/m (1200 to 12000 Oe). In consideration of that when this magnetic particle is applied to a magnetic recording medium, so that a recording head can be compatible with the magnetic recording medium, this coercive force is more preferably in the range of 95.5 to 398 kA/m (1200 to 5000 Oe).

[0146]

The particle size of the magnetic particle is preferably in the range of 1 to 100 nm, more preferably in the range of 3 to 20 nm, and most preferably in the range of 3 to 10 nm.

< < Magnetic recording medium > >

A magnetic recording medium of the present invention contains magnetic particles in its magnetic layer and the magnetic particles are those manufactured by the above-described method of manufacturing a magnetic particle of the present invention.

[0147]

As the magnetic recording medium, magnetic tapes such as a video tape and a computer tape, magnetic disks such as a floppy(R) disk and a hard disk, etc. can be enumerated. As described above, in a case where alloy particles (an alloy-particle-containing solution) are applied to a backing and changed to magnetic particles by performing annealing treatment, this layer constituted by magnetic particles can be used as a magnetic layer. Furthermore, in a case where alloy particles on a backing are not subjected to annealing treatment and instead magnetic particles are formed by performing annealing treatment in the state of a particle, the magnetic particles are kneaded by use of an open kneader, a three-roll mill, etc. and then finely dispersed by use of a sand grinder etc. thereby prepare an application solution, and this solution is applied to a backing by a publicly known method to form a magnetic layer.

[0148]

The thickness of a prepared magnetic layer, which depends on the types of magnetic recording media to be applied, is preferably in the range of 4 nm to 1 μm and more preferably in the range of 4 nm to 100 nm.

[0149]

A magnetic recording medium of the present invention may contain other layers as required in addition to the magnetic layer. For example, in the case of a disk, it is desirable to provide a further magnetic layer and a nonmagnetic layer on the surface on the side opposite to the magnetic layer. In the case of a tape, it is desirable to provide a back layer on the surface of an insoluble backing on the side opposite to the magnetic layer.

[0150]

Furthermore, by forming a very thin protective film on the magnetic layer thereby to improve wear resistance and in addition, by applying a lubricant to this protective film thereby to improve slip properties, it is possible to obtain a magnetic recording medium having sufficient reliability.

[0151]

As the materials for the protective film, it is possible to enumerate oxides such as silica, alumina, titania, zirconia, cobalt oxide and nickel oxide, nitrides such as titanium nitride, silicon nitride and boron nitride, carbides such as silicon carbide, chromium carbide and boron carbide, carbons such as graphite and amorphous carbide, etc. However, hard amorphous carbon generally called diamond-like carbon is especially desirable.

[0152]

A protective carbon film constituted by carbon, which is a very thin film having sufficient wear resistance and does not easily cause sticking to sliding members, is suitable as a material for the protective film.

[0153]

In hard disks, it is general practice to adopt the sputtering method as a method of forming a protective carbon film. In products which require continuous film forming as with a video tape, however, many methods by which plasma CVD having a higher film forming speed is used have been proposed. Therefore, it is desirable to apply these methods.

[0154]

It has been reported that in the plasma injection CVD (PI-CVD) method among others, the film forming speed is very high and that a good protective carbon film which is hard and has few pinholes is obtained (for example, in Japanese Patent Application Laid-Open No. 61-130487, Japanese Patent Application Laid-Open No. 63-279426 and Japanese Patent Application Laid-Open No. 3-113824).

[0155]

The Vickers hardness of this protective carbon film is preferably not less than 1000 kg/mm^2 and more preferably not less than 2000 kg/mm^2. It is preferred that the crystal structure of this protective carbon film be an amorphous structure and that the protective carbon film be electrically nonconductive.

[0156]

In a case where a diamond-like carbon film is used as a protective carbon film, this structure can be confirmed by a Raman scattering spectroscopic analysis. That is, when a

diamond-like carbon film is measured, this structure can be confirmed by that a peak is detected in the range of 1520 to 1560 cm^{-1}. When the structure of a carbon film deviates from a diamond-like structure, the peak detected by a Raman scattering spectroscopic analysis deviates from the above range and, at the same time, the hardness as a protective film also decreases.

[0157]

As the carbon materials for forming this protective carbon film, it is desirable to use carbon-containing compounds, including alkanes such as methane, ethane, propane and butane, alkenes such as ethylene and propylene, and alkynes such as acetylene. Furthermore, a carrier gas such as argon and an additive gas for improving the film quality, such as hydrogen and nitrogen, can be added as required.

[0158]

If the film thickness of the protective carbon film is too thick, this results in the worsening of the electromagnetic transducing performance and a decrease in the adhesion to a magnetic layer. If this film thickness is too thin, the anti-grindability becomes insufficient. Therefore, the film thickness is preferably in the range of 2.5 to 20 nm and more preferably in the range of 5 to 10 nm.

[0159]

Furthermore, in order to improve the adhesion of this protective film to the magnetic layer, which provides a substrate, it is desirable to improve the surface quality by etching the surface of the magnetic layer beforehand by using an inert gas or by exposing the surface to reactive gas plasmas of oxygen etc.

[0160]

In order to improve the electromagnetic transducing performance, the magnetic layer may be of a multi-layered structure or have a publicly known nonmagnetic substrate layer or intermediate layer under the magnetic layer. In order to improve the travel endurance and corrosion resistance, it is desirable to apply a lubricant or a rust preventive agent to the above-described magnetic layer or protective film as described above. As a lubricant to be added, it is possible to use publicly known hydrocarbon-base lubricants, fluorine-base lubricants, extreme-pressure additives, etc.

[0161]

As the hydrocarbon-base lubricants, it is possible to enumerate carboxylic acids such as stearic acid and oleic acid, esters such as butyl stearate, sulfonates such as octadecyl sulfonate, phosphate esters such as monooctadecyl phosphate, alcohols such as stearyl alcohol and oleyl alcohol, amides carboxylate such as amide stearate, amines such as stearylamine, etc.

[0162]

As the fluorine-base lubricants, it is possible to enumerate lubricants in which part or all of the alkyl groups of the above-described hydrocarbon-base lubricants are substituted with fluoroalkyl bases or perfluoro polyether bases.

[0163]

The perfluoro polyether bases are a perfluoro methylene oxide polymer, a perfluoro ethylene oxide polymer, a perfluoro-n-propylene oxide polymer $(CF_2\ CF_2\ CF_2\ O)_n$, a perfluoro isopropylene oxide polymer $(CF\ (CF_3)\ CF_2\ O)_n$ or copolymers of these polymers

[0164]

Furthermore, compounds in which polar functional groups, such as a hydroxyl group, an ester group and a carboxyl group, are present in the terminal or molecules of the alkyl groups of a hydrocarbon-base lubricant, are effective in reducing the frictional force and hence suitable.

[0165]

The molecular weight of these substances is in the range of 500 to 5000 and preferably in the range of 1000 to 3000. When the molecular weight is less than 500, the volatility may sometimes be high and the lubricity may sometimes be low. When the molecular weight exceeds 5000, the viscosity increases and a slider is apt to be adsorbed by a disk, with the result that travel stops and head crushes may sometimes become apt to occur.

[0166]

This perfluoro polyether is commercially available under brand names such as FOMBLIN of Aujimond and KRYTOX of DuPont.

[0167]

As the extreme-pressure additives, it is possible to enumerate esters phosphate such as trilauryl phosphate, esters phosphite such as trilauryl phosphite, esters trithiophosphite such as trilauryl trithiophosphite and esters thiophosphate, sulfur-base extreme-pressure agents such as dibenzyl disulfide, etc.

[0168]

The above-described lubricants are used singly or in combination. In applying these lubricants to the magnetic layer or the protective film, the lubricants are solved in an organic solvent and applied by the wire bar method, the gravure coating method, the spin coating method, the dip coating method, etc. or the lubricants are caused to adhere by the vacuum evaporation method.

[0169]

As the rust preventive agents, it is possible to enumerate nitrogen-containing heterocycles, such as benzotriazole, benzoimidazole, purine and pyrimidine, derivatives obtained by introducing alkyl side chains etc. into the mother nuclei of these heterocycles, nitrogen- and sulfur-containing heterocycles, such as benzothiazole, 2-mercaptonbenzothiazole, tetrazaindene cyclic compounds and thiouracil compounds, and derivatives of these heterocycles.

[0170]

As described above, when the magnetic recording medium is a magnetic tape etc., a back coat layer (a backing layer) may be provided on the surface of the nonmagnetic backing where the magnetic layer is not formed. The back coat layer is a layer which is provided by applying a paint for forming a back coat layer, which is obtained by dispersing granular components, such as an abrasive material and an antistatic agent, and a binder in a publicly known organic solvent, to the surface of the nonmagnetic backing where the magnetic layer is not formed.

[0171]

As the granular components, it is possible to use various kinds of inorganic pigments and carbon black. As the binders, resins such as cellulose nitrate, phenoxy resin, vinyl chloride resin and polyurethane can be used singly or in combination.

[0172]

Furthermore, a publicly known adhesive layer may be provided on the surface to which the alloy-particle-containing solution is applied and the surface on which the back coat is formed.

[0173]

When the cut-off value is 0.25 mm, the centerline average roughness of the surface of a magnetic recording medium thus manufactured is preferably in the range of 0.1 to 5 nm and more preferably in the range of 1 to 4 nm. This is because providing a surface having an excellent smoothness is desirable for a magnetic recording medium for high-density recording.

[0174]

As a method of obtaining such a surface, it is possible to mention a method which involves performing calendaring treatment after the formation of the magnetic layer. Also, burnishing treatment may be performed.

[0175]

A magnetic recording medium thus obtained can be appropriately punched by use of a punching machine or cut to a desired size by use of a cutting machine so that it can be used.

[0176]

[Advantages of the Invention]

As described above, according to the method of manufacturing a magnetic particle of the present invention, it is possible to prepare an alloy particle which satisfies all the requirements for a micro size, monodispersibility and transformation facilitativeness in the alloy particle preparation step and, therefore, it is possible to manufacture a magnetic particle of good performance.

[0177]

Furthermore, a magnetic recording medium of the present invention, which contains the magnetic particle manufactured by the invention in a magnetic layer, has lower noise level and high-quality performance of high recording density.

[Brief Description of the Drawings]

[Figure 1]

A desirable conceptual diagram showing the construction of a mixing and reaction device for carrying out the ultrahigh-speed stirring method in the alloy particle preparation step in a method of manufacturing a magnetic particle of the present invention.

[Figure 2]

A desirable conceptual diagram showing the construction of a mixing and reaction device for carrying out the microgap mixing method in the alloy particle preparation step in a method of manufacturing a magnetic particle of the present invention.

[Figure 3]

A sectional view of the mixing and reaction device of Figure 2 which is laterally cut.

[Figure 4]

A sectional view to explain an example of a modification of the mixing and reaction device for carrying out the microgap mixing method.

[Figure 5]

A sectional view to explain another example of a modification of the mixing and reaction device for carrying out the microgap mixing method.

[Figure 6]

A desirable conceptual diagram showing the construction of a mixing and reaction device for carrying out the in-tank mixing method in the alloy particle preparation step in a method of manufacturing a magnetic particle of the present invention.

[Figure 7]

An enlarged view of a mixer in the mixing and reaction device of Figure 6.

[Figure 8]

An explanatory drawing to explain a flow regulating valve in the mixer of Figure 7.

[Figure 9]

An explanatory diagram to explain a conventional mixing and reaction device.

[Description of Symbols]

1 ···conventional mixing and reaction device, 2 ···tank, 3 ···motor, 4 ···transmission device, 5 ···stirring vane, 6 ···temperature control device, 7 ···sealing lid, 8 ···introduction pipe, 9 ···discharge pipe, 10 ···mixing and reaction device for achieving ultrahigh-speed stirring method, 12 ···mixer, 14 ···high-speed stirrer, 16 ···rotary shaft, 20 ···bearing, 24 ···stirring vane, 26···first preparation tank, 28 ···second preparation tank, 30, 32 ···stirrer, 34, 36 ···valve 38, 40 ···addition pipe, 42 ···discharge pipe, 50 ···mixing and reaction device for achieving microgap mixing method, 52 ···mixer, 54 ···rotary drum, 56 ···rotary shaft, 58 ···bearing 62 ···microgap, 64 ···first preparation tank, 66 ···second preparation tank, 68, 70 ···stirrer, 72, 74 ···valve, 76, 78 ···supply pump, 80, 82 ···supply pipe, 84, 86 ···supply port, 88 ···discharge port, 90 ···discharge pipe, 92 ···flat disk, 120 ···mixing and reaction device for achieving in-tank mixer method, 122 ···reaction vessel, 124 ···circular opening, 126 ···mixer, 128, 130 ···addition pipe, 132 ···rotary shaft, 134 ···upper stirring vane, 136 ···lower stirring vane, 140 ···flow regulating valve, 142 ···valve main body, 144 ···valve casing, 146 ···valve plate, 148 ···inlet chamber, 150 ···valve chest, 152···inlet port, 154···outlet port, 156 ···valve rod

Application Number: 2003-059045
Application Date: March 5, 2003

[Title of the Document] Abstract

[Abstract]

[Problem to be Solved]

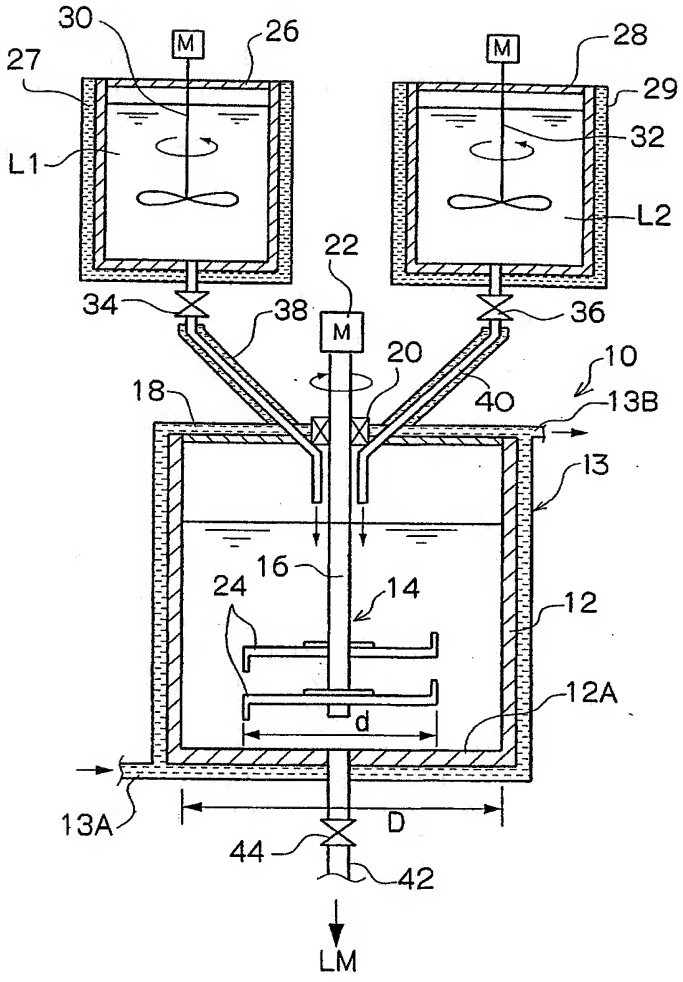
To prepare an alloy particle which satisfies all the requirements for a micro size, monodispersibility and transformation facilitativeness in the alloy particle preparation step.

[Means for Solution]

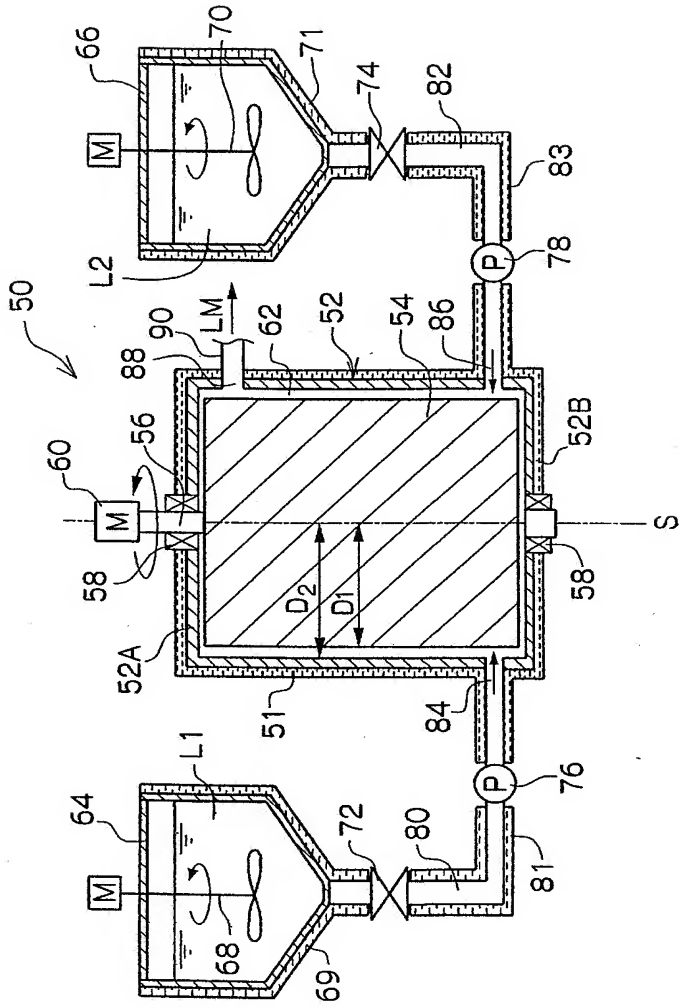
There is provided a method of manufacturing a magnetic particle, which comprises: an alloy particle preparation step of preparing an alloy particle capable of forming a CuAu type or Cu_3Au type hard magnetic ordered alloy phase and a magnetic particle formation step of forming the CuAu type or Cu_3Au type magnetic particle from the alloy particle prepared in the alloy particle preparation step; wherein in the alloy preparation formation step, by using a mixing and reaction device 10 which has a stirring vane rotating at a high speed in the interior of a mixer 12, solutions L1 and L2 for preparing the alloy particle are supplied to the interior of the mixer 12, where the solutions L1 and L2 are mixed together and caused to react with each other by a liquid phase process so that the peripheral speed in a leading end portion of the stirring vane is not less than 10 m/second.

[Selected Drawing] Figure 1

【Title of the Document】 Drawings
【Fig. 1】



【Fig. 2】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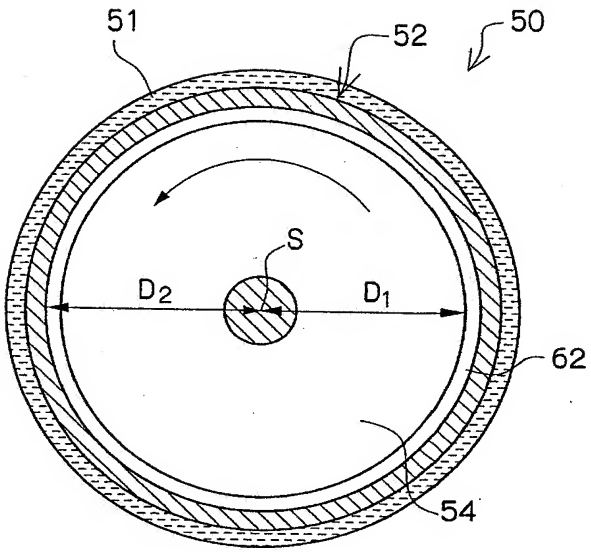
84
86
88
90
L1
L2
LM
M
P
S
D1
D2

【Fig. 3】



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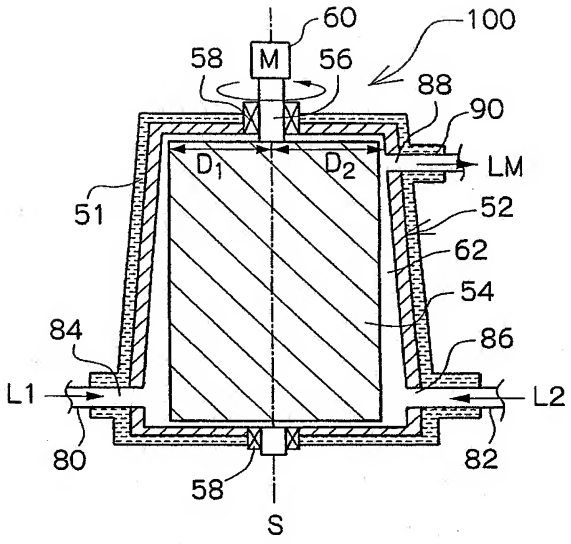

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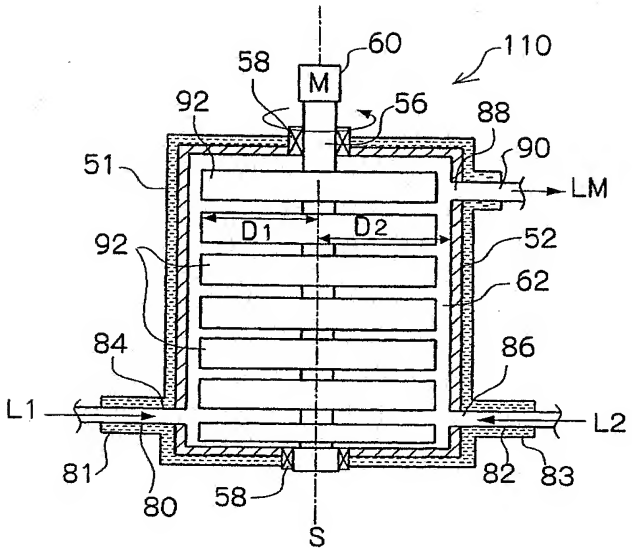
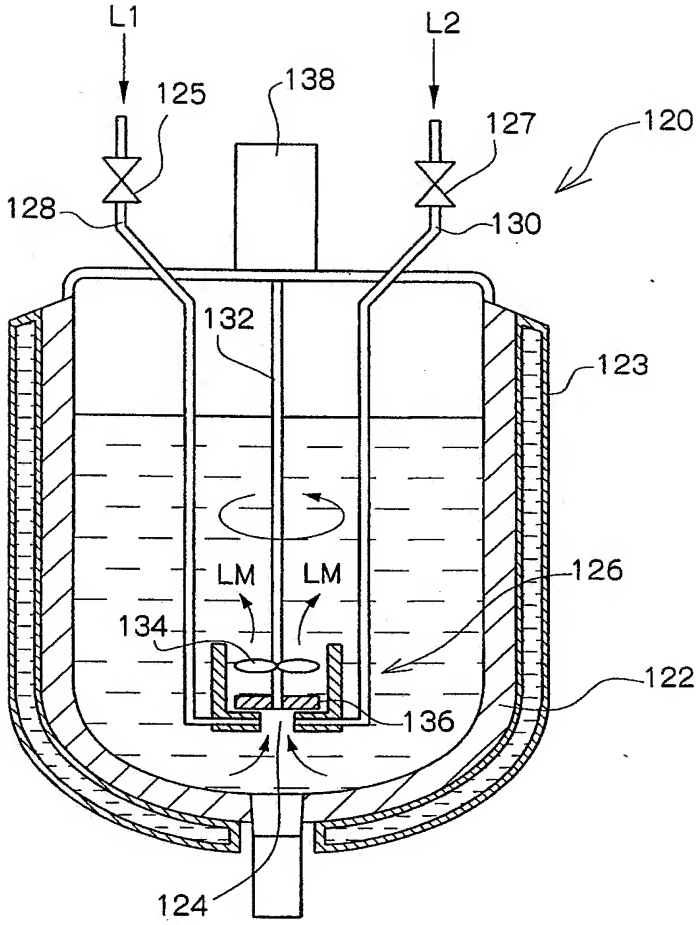
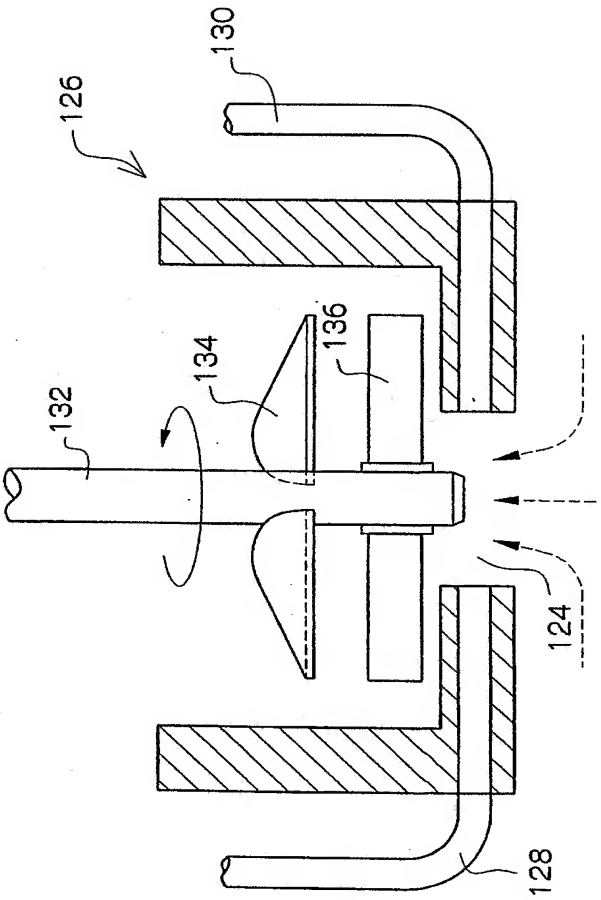


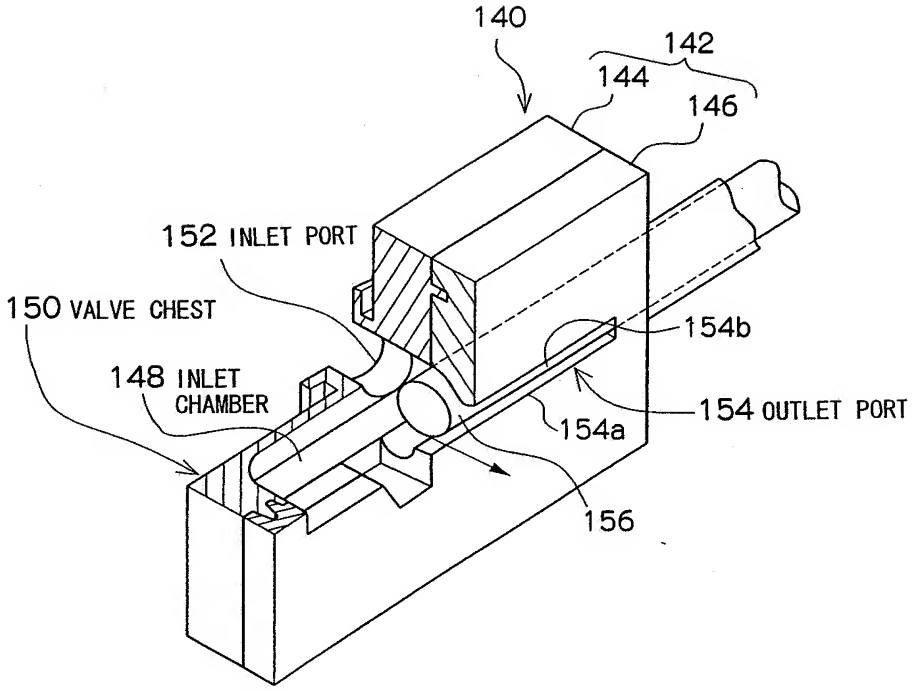
【Fig. 6】



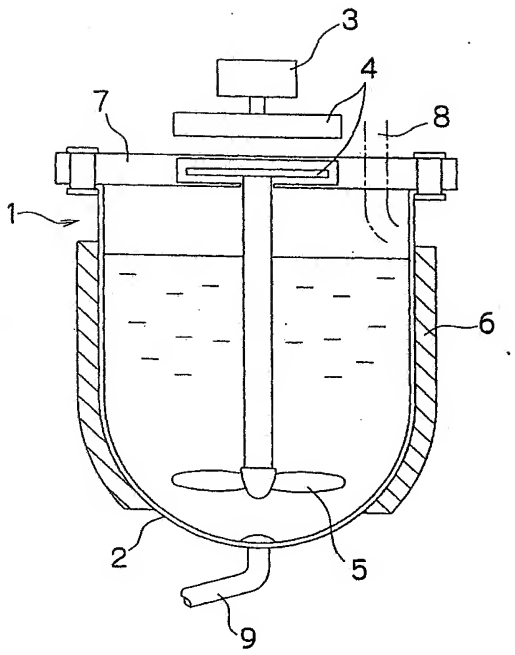
【Fig. 7】



【Fig. 8】


140
142
144
146
152 INLET PORT
150 VALVE CHEST
154b
148 INLET CHAMBER
154 OUTLET PORT
154a
156

【Fig. 9】



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[Name or Designation]	Fuji Photo Film Co. Ltd.
[Agent]	
[Identification Number]	100083116
[Patent Attorney]	
[Name or Designation]	Kenzo Matsuura
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[Title of Amended Document]	Specification
[Amended Item]	0107
[Method of Amendment]	Modification
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[Amendment 2]	
[Title of Amended Document]	Specification
[Amended Item]	0162
[Method of Amendment]	Modification
[Content of Amendment]	2
[Amendment 3]	
[Title of Amended Document]	Specification
[Amended Item]	0163
[Method of Amendment]	Modification

[0107]

Incidentally, when a zero-valent metallic compound, such as Fe carbonyl, is used as the material for a base metal, it is unnecessary to use a reductant for the base metal.

[0162]

As the fluorine-base lubricants, it is possible to enumerate lubricants in which part or all of the alkyl groups of the above-described hydrocarbon-base lubricants are substituted with fluoroalkyl bases or perfluoro polyether bases.

[0163]

The perfluoro polyether bases are a perfluoro methylene oxide polymer, a perfluoro ethylene oxide polymer, a perfluoro-n-propylene oxide polymer $(CF_2\ CF_2\ CF_2\ O)_n$, a perfluoro isopropylene oxide polymer $(CF\ (CF_3)\ CF_2\ O)_n$ or copolymers of these polymers